A STUDY ON EFFECTIVE THERMAL CONDUCTIVITY OF EPOXY- Al_2O_3 COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology

in

Mechanical Engineering

(Thermal Engineering)

By

KSHITIJ CHANDRA

213ME3427

DEPARTMENT OF MECHANICAL ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

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ROLL NO-213ME3424

Under the guidance of

PROF. ALOK SATAPATHY

DEPARTMENT OF MECHANICAL ENGINEERING

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CERTIFICATE

This is to certify that the thesis entitled, "**A STUDY ON EFFECTIVE THERMAL CONDUCTIVITY OF EPOXY- Al_2O_3 COMPOSITES**" submitted by Mr. KSHITIJ CHANDRA in partial fulfilment of the requirement for the award of Master of Technology Degree in Mechanical Engineering with specialization in Thermal Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter included in the thesis has not been submitted to any other University / Institute for the award of any degree or diploma.

Date

Prof. Alok Satapathy
Associate Professor
Department of Mechanical Engineering
National Institute of Technology Rourkela
Rourkela– 769008

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Date

Place -NIT Rourkela

Kshitij Chandra

213me3427

CONTENTS

ABSTRACT

The motive of our present work is to estimate the thermal conductivity of the epoxy based polymer composite filled with aluminium oxide or alumina (Al_2O_3) particulate for the purpose of exploring its use in the field of microelectronic industry. In microelectronic industry there is too much demand of low weight and high thermal conductivity, low electrical conductivity materials for the purpose of excellent heat dissipation through the component so that product life can be increased. Here we have worked on low to high volume percentage of the filler up to the mark of 0 to 26.18% to calculate their corresponding thermal conductivity, and tried to achieve our goal. First suitable mathematical model has been developed using sphere- in –cube models having face centered arrangement of spherical particle, then derived the expression for thermal resistance of that model and finally with the help of thermal resistance we calculate the thermal conductivity of that model with different volume percentage of the filler material. After doing all that verified our result obtained from mathematical model with the experimental results for each specimen of different volume fraction. Results are also compared with the various available mathematical model like Aggarwal and Satapathy model, rules of mixture, Maxwell model, Raleigh model, Lewis and Neilson model etc. As there is continuous miniaturization in the field of the microelectronics industry demanded need of having better heat dissipation along with low high electrical resistivity. The material is selected to fulfill the requirement of the demand. Here epoxy is used as the matrix material whereas aluminium oxide is used as filler materials.

Key words*: Polymer Composite, volume fraction, Thermal Conductivity, filler material, mathematical model*

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CHAPTER 1

INTRODUCTION

1.1 Introduction to composite material

Composite materials can be defined as a material consists of two materials of different phases such as reinforcing phase and matrix phase. Reinforcing phase is in the form of fiber sheets or materials which are embedded in another material termed as matrix phase. Matrix material used to transfer stresses among composite material and protect composite from different damage such as mechanical and environmental damages whereas the fiber particles are used for the purpose of improvement of mechanical properties which is strength, stiffness etc. hence we can say like that composite material are combination of two different material which are different in physical and chemical composition and not soluble in each other. Our purpose of doing all like that is to take advantage of required properties of each materials and obtain better mechanical properties from the previous both. As there is the progressive development happens in the history of the composite material it replaces more of the conventional material used earlier as it is superior in the form of light weight, excellent strength to weight ratio, good tensile strength, high toughness, high creep resistant and it can be made as per our requirement. Generally in the composite material reinforced material are of low density with high strength whereas the matrix material properties are ductile of tough in nature. As composites are fabricated perfectly it take strength and toughness from the reinforced and matrix material respectively and there combination give us desired properties which are not available in any conventional material. The properties of the composite depend upon the arrangement, amount, and type of fiber and or particle which are reinforced in the matrix resin.

The concept of composite material is natural, it can be understand as wood is the combination of cellulose fiber and polysaccharide lignin. In the two phase of composite material one phase is generally discontinuous, stronger and stiffer termed as the reinforcement on the other hand another phase is continuous with comparative less strength and stiffer termed as matrix. The properties, what we obtained by doing all these process depends upon the various condition such as the distribution of the reinforced particle and its constituents with geometry of the arrangement and one other biggest parameter to determines properties is the volume and weight percentage of the reinforcement particle. Homogeneity or uniformity of the material system also depends upon the distribution of the reinforced particle, as we all know the importance of the homogenous materials for the purpose the strength in equally distributed load, failure of achievement of homogeneity leads to failure of the material from the weak areas or where there is a lack of particle reinforcement. The anisotropy of the composite material is very much influenced by the geometry and orientation of the reinforced particle.

The selection of composite for various purposes, phases of the composite system plays a very important role. The composite material used for the purpose of low to medium performance, reinforcement generally occur in the form of short fiber and particle provide some stiffening and strengths up to the moderate value whereas the matrix is specially used for the load bearing as per requirement of the mechanical properties. Composite which are used for the high performance, the is too much requirement of the continuous fiber reinforcement which determines the strength and stiffness in this case matrix phase of the composite provides support and protection to the sensitive fiber and it also transfer stress from one fiber to other fiber. Although the interface is negligible however it plays important role in protecting composite from the failure.

1.2 Different types of composite materials

Composite material can be classified in to the different category on the basis of their constituents. On the basis of matrix material it can be divided in following three categories:

a) **Metal matrix composite (MMC)**

b) **Polymer matrix composites (PMC)**

c) **Ceramic matrix composite (CMC)**

a) Metal matrix composite (MMC)

Composite material where metallic material is used as the matrix of the composite is termed as metal matrix composite. It has too much advantage comparative to the monolithic metal such as good specific strength, specific modulus, lower coefficient of thermal expansion also it has better properties at elevated temperature. Due to all these unexpected excellent properties it can be used for wide range of Varity of application some of those where it is frequently used are combustion chamber nozzle of space shuttle and rocket, housing, heat exchanger, tubing, structural members, cables etc.

b) Polymer matrix composite:

This types of matrix is most commonly and extensively used among all the metal matrix composite. These are the two strong reason fulfill above statement. First polymers mechanical properties are insufficient for the structural purposes. In general strength and stiffness of polymer are low compared to metals and ceramics. These all difficulties are removed by the reinforcing other materials with polymers. Second reason is the processing or fabrication of polymer matrix composite are simpler than other as it not demands application of high pressure and also does not require high temperature. Also the equipment involves in the processing of polymer matrix composite are simpler. Because of all these the advantage it can be developed easily and became very popular for the use of structural application. The overall properties of these composites are far superior than individual but is not as much brittle as the ceramics.

Polymer composites are broadly classified in to two categories based on the particle of enforcement which are named as the fiber reinforced polymer (FRP) and particle reinforced polymer (PRP). This classification are nicely explained below with the help of figure 1.1

Polymer composite classification:

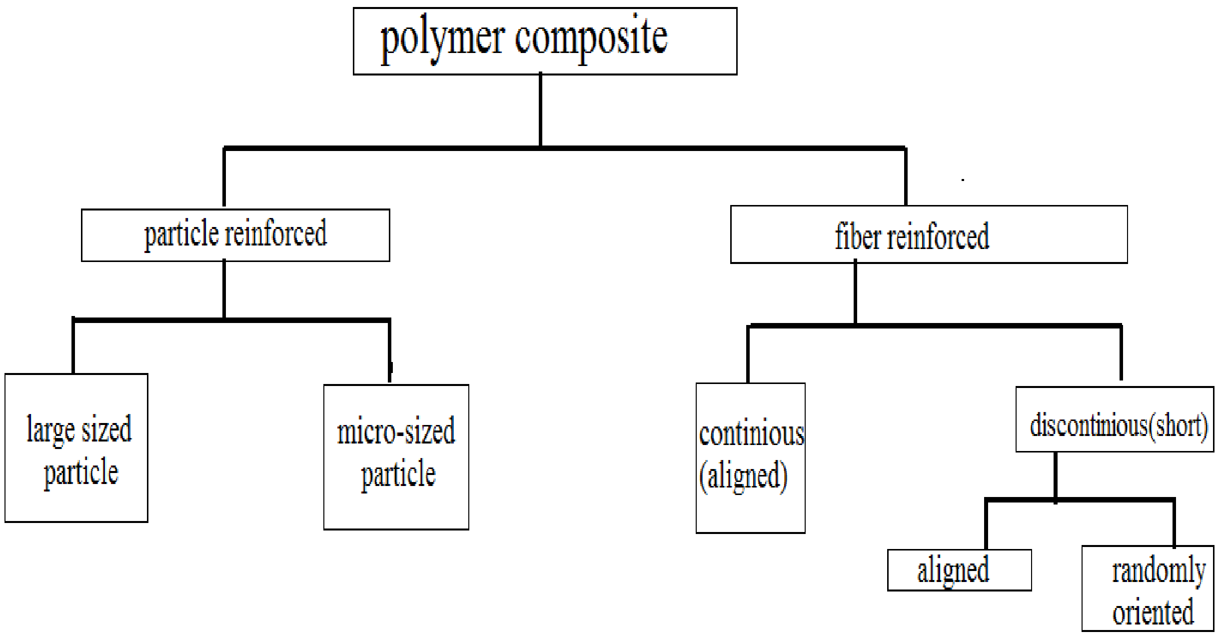

Fig 1.1 Classification of polymer composite based on reinforcement

Fiber reinforced polymer composite:

Fiber reinforced composite contains matrix and fiber. matrix materials commonly used in fiber reinforced composite are epoxy, vinyl ester, phenolic resin, polyurethane, polyester etc. among all these matrix polyester is most commonly used, epoxy is better options as it has higher adhesion and less shrinkage than polyester but it is second most widely used resin due to its high cost. Most commonly used fiber reinforcing material is asbestos, beryllium oxide, glass fiber, beryllium carbide, molybdenum, carbon/graphite fibers, natural fiber, polyamide etc. fiber are mainly used for the source of strength while matrix function is transfer stress among reinforcing fiber and fix all the fiber together in well required shape. Fiber carries load among the direction of longitudinal axis. Sometimes filler materials are also inserted for the purpose of to impart special properties, smoothness in manufacturing process, and to reduce production cost.

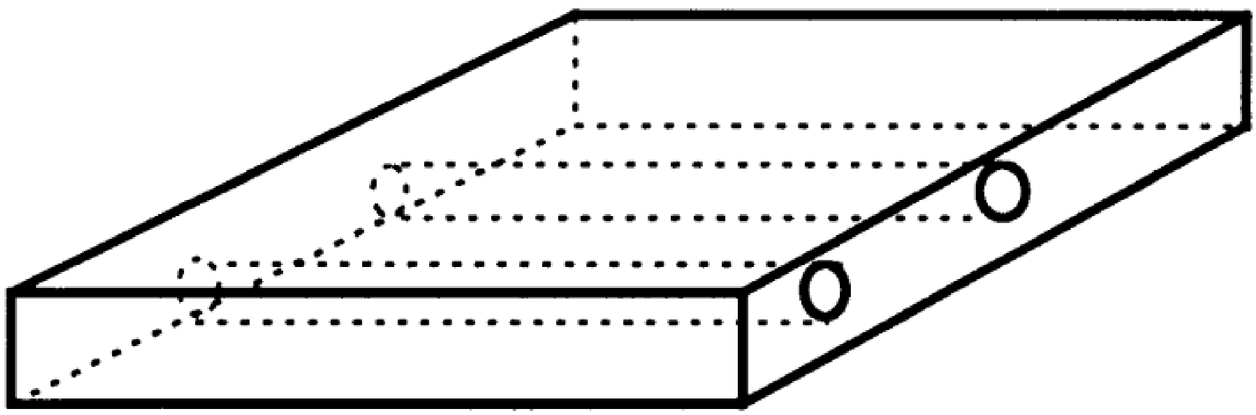

Fig 1.2 Fiber reinforced polymer composite

Particle reinforced polymer composite:

Ceramics, metal powder (aluminum oxide, titanium oxide etc.), small mineral particle, amorphous materials, carbon black etc. are used as particle for reinforcing. These particles used for decrease the ductility and increase the modulus of the composite it also reduces the cost of the matrix. Particles like ceramic and glasses has properties like high melting temperature, high strength low density corrosion and wear resistance. Mostly ceramics act as good thermal and electrical insulator. Some ceramic has magnetic properties, some has piezoelectric, some are superconductor at very low temperature in spite of all these properties its major drawback as brittle in nature. An excellent example of particle reinforced composite is automobile tires, whose matrix material is poly isobutylene elastomeric polymer and reinforcement of carbon black particles.

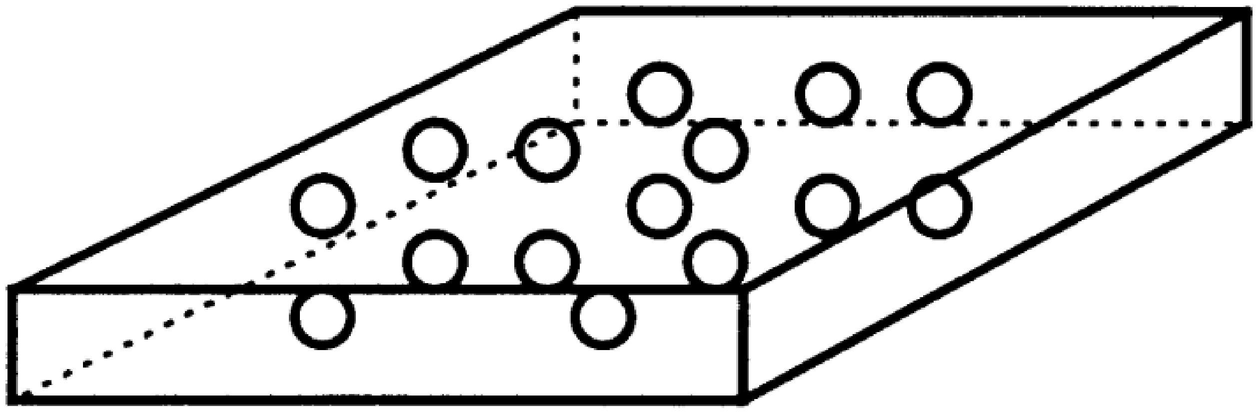

Fig 1.3 Particulate reinforced polymer composite

c) **Ceramic matrix composite**:

Matrix material used in the ceramic composite is ceramic with various commercially used binders. One of the superior advantage that can be achieved by the ceramic matrix composite is toughness can be increased reasonably. Naturally it is found that there is concomitant improvement by ceramic matrix composite in stiffness and strength.

1.3 Background and motivation:

Excellent life of micro-electronics products depends upon their physical properties as well as their thermal characteristic such as heat conduction capability of the component used. Packaging of Micro-electronics is the superior requirement in the development of the electronic technology. As due to advancement in this industry there is requirement of large scale integration of various components in the single circuit which causes results in large scale heat generation hence there is tremendous probability of failure of the product. Viewing all these difficulties, large scale heat dissipation is the urgent need to avoid the overheating of the product. Material used for packaging of the micro-electronics products also requires low relative permittivity along with reduction in dielectric loss with addition of all these properties there is also requirement of low coefficient of thermal expansion.

Use of high cost embedded heat sinks in encapsulated devices traditionally addresses the heat dissipation problem in addition to all these there is also fear of thermal cracking, due to thinner configurations of package utility is also limited. Having good mechanical, electrical properties of the ceramic and various polymer leads to the great demand in the form of packaging materials in microelectronics industry [1]. However, common packaging polymers are suffering from the drawback of low thermal conductivities incorporate this unable in heat dissipation, high thermal coefficient of these polymer leads to thermal failure. Incorporation of all these drawback continues the exploring of some noble material. When these polymer filled with particulates having good thermal conductivity leads a cost effective way to counterfeit with all thermal issues [2]. Composites definitions not widely accepted however it an artificially made multiphase materials having properties better than constituents materials. Its property also depends upon the shape and size of the reinforced particles.

1.4 Introduction to the research topic

The present research discuss about the search of some composite material which are the urgent requirement in the field of microelectronics industries. As in this field there is day by day drastically miniaturization of electronics devices which requires some material which have excellent thermal conductivity along with light weight, high electrical resistivity so that there is possibility of solutions of the optimal heat dissipation through the component of electronic devices. This research is in the direction of accept challenge of requirement and give the solutions i.e. fabrication of composite which can fulfill the above requirement. Thermally conductive filler can be inserted in to the polymer with suitable volume fraction can increase the thermal conductivity by keeping the other parameter in agreement with requirement, This concept is the pillar of the present work.

In present research for the estimation of effective thermal conductivity, a mathematical model using sphere-in cube arrangement in face centered cubic manner has been developed which was based on the law of minimum thermal resistance. Further by varying the filler (Al_2O_3) volume percentage calculate the different thermal conductivity value with different volume fraction (3.27, 7.75, 13.4, and 26.18%). These results are also compared with the various existing model. For the purpose of validation of developed model there was an investigation done by measuring thermal conductivity of fabricated composites experimentally by using the Unitherm^{TM} Model 2022. For the fabrication of the composites epoxy LY 556 resin is used as a matrix material which generally chemically belongs to 'epoxide' family. It is most common used thermoset polymer. It is used with the hardener HY 951. Whereas alumina particle is chosen as the filler material.

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CHAPTER 2

LITERATURE REVIEW

History of composite materials came in to existence since 1930's by seeing its various advantage it gets drastic popularity. Since then there is continuous development occurring year by year in this field. This topic of thesis contains study of some previous paper published in various journal regarding the continuous development of composite material on various aspect.

2.1 Particulate filled polymer matrix composite

Polymer matrix composites manufactured as per requirement through adding filler in matrix material. These fillers function is to enhance the various properties such as wear resistance and hardness, cost reduction, control of thermal expansion, density control. In recent time ceramic or metal particles used as hard filler whereas glass is used as the fiber filler to improve the wear resistance up to two to three times [3]. In various application of industry such as heater, electrodes, thermal durability at high temperatures polymer matrix composite with metal particulate filler are used [4]. These engineering composite have large applications due to their ease of fabrication, cost reduction, high corrosion resistance and low density [5]. Similarly inorganic materials as ceramic are the subject of research in last two decades for its advantage as cost reduction and improvement in stiffness [6]. Bonner [7] reported high filler content up to 20% volume fraction is required to achieve above properties but it also has some detrimental properties like difficulties in fabrication, density etc. silica particles when used to form composite it improves considerable change in thermal , electrical and mechanical properties of fabricated composite [8]. In addition to all these past year many researchers shows interest in reduction of particle size and effect of single particle, how bring the changes in the various properties of the polymer matrix composite [9-11]. Yamamoto et al. [12] shows silica particles shape and structure affect mechanical properties such as fracture properties, fatigue resistance, and tensile strength. Nakamura et al. [13] reported change in shape and size, in the form of increase in surface area increases mechanical properties as strength and fracture toughness. Also shows particle matrix adhesion increases the fracture toughness. Yuan et al. [14] and Ng et al. [15] discussed the effect of micron size particle and Nano-particle on the properties of the polymer matrix composite.

Two main theory theories first filler theory and second as mastic (filler matrix system) theory used in formulation and production of composite. According to filler theories [16] when particle size and distribution allow maximal packing of filler particle then optimal properties of composites are achieved where is in the case of mastic theory [17] matrix material effect as coating on particulate of optimal thickness. Other parameter which affects the mechanical behavior of composites is wetting of the filler by resin and adhesion of the two components. Srivastava and shembekar [18] researched about the fracture toughness of the epoxy resin and concluded that as there is addition of fly ash particles as filler there is some improvement in the toughness. It also affects the tensile characteristic according to size and interfacial bonding. Das et al. [19] investigated about the cenosphere filled polypropylene structural and dynamic mechanical properties and obtained there is enhancement in damping properties whereas tensile strength and impact strength decreased with increase in percent of filler.

2.2 Thermal Conductivity of Polymer Matrix Composites

Sufficient amount of literature on the transport of heat in polymers can be found in research papers of Hansen and Ho [20], Knappe and Hennig[21], Chou and Young[22], Peng and Landel[23], etc. Tavman [24], by altering polymers' orientation of molecules, successfully gave a condition for their non-isotropic heat conduction. Griesinger et al. [25] recently have proved that by fixing orientation ratio at 50, polyethylene thermal conductivity can be enhanced from 0.35-50 W/m-K.

Augmentation of polymer's thermal conductivity can also be achieved by adopting a more practical method, that is, by including thermally conductive fibers or particles into it. There exists many research papers that report the augmentation of polymer's thermal conductivity by encapsulating conductive fillers. Majority of these works are experimental [26-28].

Metals are widely introduced as fillers in composites of fillers since they are capable of transferring heat by conduction effectively. Sofian et al. [29] observed the influence of different metal powders like zinc, bronze, iron and copper on specific heat diffusivity and conductivity of denser materials. Tavman [30] incorporated filler as aluminium powders and observed high-density polyethylene thermal conductivity. Boudenne et al. [31] presented their views on aluminium/polypropylene composites thermal conductivity. Furthermore, due to its higher conductivity, silver, as a filler material, can be effectively used to augment thermal conductivity of higher density materials.

Carbon-based fillers have the highest potential to be effective fillers, as they have high low density and higher thermal conductivity. Some popularly recognized carbon-based fillers are carbon-black, carbon fiber and graphite. The filler that has the best thermal conductivity is graphite because of its low cost and good thermal conductivity [32]. Higher thermal conductivity can be achieved by employing single graphene sheet with graphite [33]. Han and Fina [34] conducted studies on polymer composites containing carbon nanotubes and their influence on modified thermal conductivity of them. In electronic devices, features like high electrical conductance and thermal conductivity are required. Such features can be achieved by using carbon-based and metallic fillers.

Boudene et al. [35] observed the influence of two distinct particle sizes of aluminium filler and reported that significant higher thermal conductivities can be achieved with particles sizes of larger dimensions. This results from the presence of more stable conductive pathways in the composite.

Zhou et al. [36] reported similar behavior. Boudenne et al. [37] reported opposite behaviors when they experimentally found higher thermal conductivities with smaller sized particles. A study of the influence of filler particles interconnectivity on the thermal conductivity of composites was experimentally determined by Weidenfellar et al. [38]. Veyret et al. [39] numerically studied the effect of fibrous or granular reinforced composite materials while Kumlutas and Tavman [40] constructed a numerical model in which filler materials accounted for 10 % of volume of the composite. Their results closely matched with that of experimental ones. Nayak et al. [41] have recently predicted the thermal conductivity of epoxy composite filled with pine wood with the help of finite element method. Cai et al. [42] developed a 3-dimensional FEM model to account for the prediction of PTFE composites thermal conductivity, while the fillers size distribution was taken into consideration.

2.3 Thermal conductivity models

For the prediction of effective thermal conductivity of two different composition material lots of co-relation and theoretical models are developed by various researcher in past decades. In 1873 Maxwell was the first who gave the first model for estimation of effective thermal conductivity of two phase mixture hence the Maxwell-Garnett model was the first among the all theoretical model. For the applicability of these model various articles have discussed [43, 44]. The simplest alternative for the two component composite is that the materials are arranged in either series or parallel with the direction of heat flow, the limit of thermal conductivity obtained by this arrangement gave lower and upper bound of the thermal conductivity respectively. The equation 2.1 for series and 2.2 for parallel for these two model are shown below

$$\frac{1}{k_{eff}} = \frac{1-\emptyset}{k_e} - \frac{\emptyset}{k_p} \tag{2.1}$$

$$k_{eff} = (1 - \emptyset)k_e + \emptyset k_p \tag{2.2}$$

These two equations are derived on the basis of rules of mixture (R.O.M) [45]. Another model based on based on parallel and series conduction was developed by Agari and Uno [46] this model gives the expression of thermal conductivity in following form

$$\log k_{eff} = \emptyset c_2 \log k_p + (1 - \emptyset) \log(c_1 k_e) \tag{2.3}$$

Where constant c_1, c_2 are determined by constant of order unity.

Assuming isotropic particulate reinforcement along with considering shape and orientation of filler particle a semi- theoretical model is developed by Lewis and Nielson [47] which was the modification of Halpin-Tsai two phase equation. The expression of this model looks like

$$k_{eff} = k_e \left[\frac{1+AB\emptyset}{1-BC\emptyset}\right] \tag{2.4}$$

For spherical particle,

$$A=1.5, \quad B = \frac{\frac{k_p}{k_e}-1}{\frac{k_p}{k_e}+1.5} \quad \text{and } C= 1+\emptyset\left(\frac{1-\emptyset_m}{{\emptyset_m}^2}\right)$$

Where, $\emptyset_m$= maximum packing fraction of filler (0.637)

Maxwell equation [48] gave an idea to derive the expression for k_{eff} of spherical particulates in a continual medium. The expression derived by Maxwell model for effective thermal conductivity seem like

$$K_{eff} = k_e \left[\frac{k_p+2k_e+2\emptyset(k_p-k_e)}{k_p+2k_e-\emptyset(k_p-k_e)}\right] \tag{2.5}$$

Aggarwal et al. [49] derived a mathematical correlation based on law of minimum thermal resistivity for hybrid polymer composite filled with two different particulate filler material to calculate the effective thermal conductivity which fits very approximately to the experimental value up to some extent of filler material. Expression of this model was

$$K_{eff} = \frac{1}{\frac{1}{k_e} - \frac{1}{k_e}\left(\frac{6\emptyset}{\pi}\right)^{\frac{1}{3}} + \frac{4}{k_e\left(\frac{4\pi}{3\emptyset}\right)^{\frac{2}{3}} + \left(\frac{2\emptyset}{9\pi}\right)^{\frac{1}{3}} \times 2\pi\left(k_p - k_e\right)}} \qquad (2.6)$$

2.4 Polymer matrix composite reinforced with alumina particles

There is some work when aluminium oxide was used as a filler material. The best among them was found when investigations were performed using nano particles micro-sized aggregates, since the filler was introduced in the form of a powder to the resin [50]. Since these fillers augment the thermal stability and strength of the material in addition to imparting resistance to corrosive media, hence aluminium oxide with epoxy matrix reinforcements are attractive. For the purpose of making tools in lesser volume production in an economical and rapid manner, S.Ma et al. [51] investigated some epoxy composites reinforced with particles. This method used for making tools can be applied for polymer and wax materials moldings applications. The development of epoxy matrix composites reinforced with glass fiber that were filled with alumina was proposed by S.Biswaset et al. [52] and compared the theoretical erosion model with Taguchi's experimental results. Such composites are suitable for environments involving high erosion. A.Pattnaiket et al. [53] measured abrasive of randomly oriented epoxy resin filled in aluminium oxide, pine bark dust and silicon carbide that was glass fiber reinforced. O. Asi [54] performed an experiment to measure bearing strength behavior of glass fiber reinforced composites pinned joints. He stated that with 15 % aluminium oxide, there was first decrease in bearing strength, and then there was decrease when aluminium oxide content was further decreased as a result of lower void content in previous percentage. The electrochemical and tribological corrosion of aluminium oxide polymer was determined by Y.Wangel et al. [55] and they showed a considerable enhancement in abrasive and scratch resistance for 20 % aluminium oxide when compared to the polymer coating. In matrices, when aluminium oxide nano-particles were introduced, the composite's mechanical strength considerably augmented whereas when aluminium oxide nanofibers were filled, there was increment in Young's modulus. This phenomenon was studied by B.N. Dudkinet et al. [56].

In the past, alumina filled in epoxy composites effective thermal conductivity study has been very limited and the numerical approach in this field was not even initiated. L.C.Sim et al. [57] performed experiment to find the influence of introduction of aluminium oxide and tenorite nano-particles to a UPR matrix having lesser thermal conductivity on the effective thermal conductivity of whole composite.

2.5 Knowledge gap in earlier investigations:

Although there is various investigation has to be found and published based on enhancing effective thermal conductivity of polymer composites but today also there is wide scope of improvement is required to get precious results. Some important gap in research are concluded as,

- In spite of enhancing both thermal as well as dielectric properties of material, most work are available on enhancing only thermal properties.
- No systematic report is found on the basis of alumina particulate as filler material.
- The relationship among the effective thermal conductivity with the micro-structural properties (volume fractions, aggregation of particles, distribution of particles, properties of individual components, etc.) is far from satisfactory.

In the present work there is systematic study of work based on enhancing the thermal conductivity along with care on dielectric properties with the help of epoxy-alumina composite. Study also shows the variation of composites properties with the volume fraction of the filler.

2.6 Objectives of present research

Outline of the present research are

1. Fabrication of set of alumina filled composites of different volume fraction for the purpose of maximum heat dissipation through it.
2. To develop a theoretical model for estimation of effective thermal conductivity of polymer composites reinforced with filler material.
3. Study the filler effect on volume fraction on the effective thermal conductivity of the fabricated composite by theoretically developed model and comparing it with various developed co-relation and model.
4. To validate the obtained results from the proposed mathematical co-relation through experimental measurements of polymer composites thermal conductivity fabricated with different filler concentration.
5. To study the effect of alumina particulate filler on the thermal characteristic of the fabricated polymer composites.
6. Exploring the possible use of fabricated composites.

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CHAPTER 3

MATERIALS AND METHODS

3.1 Material selection

It presents the constituents of material used in the fabrication of epoxy-alumina polymer composite

(a) Matrix material:

Epoxy LY 556 resin is used as a matrix material which generally chemically belongs to ‘epoxide’ family. The common name of this material is Bisphenol-A-Diglycidyl-Ether. Mostly epoxy are derived from the petroleum products. Plants derived composites are also available. A large number of variety of epoxy resins are commercially available for composite fabrication. Epoxy is chosen because it has low density (1.1gm/cm3) and it is most common used thermoset polymer. It is used with the hardener HY 951. It has low thermal conductivity (0.363W/m-K).

Fig.3.1 Epoxy and Hardener container

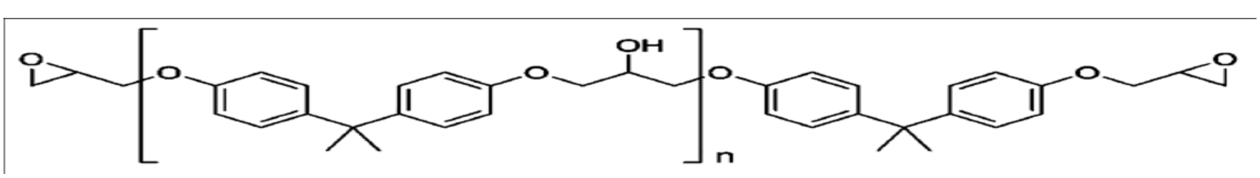

Fig 3.2: epoxy polymer resin chain.

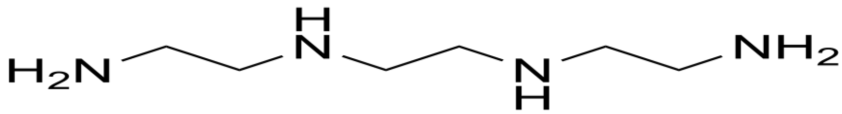

Fig. 3.3 Tri-ethylene-tetramine (hardener used for epoxy matrix)

Table3.1: Properties used for epoxy resin

Property	**Numerical value**
Density	1.1 gm/cc
Thermal conductivity	0.363w/m-k
Micro hardness	0.085GPa

(b) Filler material (Al_2O_3):

Micro-sized Al2O3 is used as a filler particulate for the preparation of thermally conductive PMCs. It is organic materials that can be exist in crystalline phase. Alumina contributes 15% of the earth's crust and is amphoteric in nature. It has strong ionic inter-atomic bonding. it is amphoteric (i.e. it can behave as both acid and base), hard, resistant to strong acid and alkali attack at elevated temperature, wear resistant, high strength and stiffness with an excellent combination of properties and a reasonable price. Alumina is light weight in nature, having high melting point, properties of electrical insulator and high thermal conductivity (35W/m-K)

Fig. 3.4: Commercially available alumina particles

Table3.2: Properties used for aluminum oxide

Properties	Numerical value
Density	3.89gm/c
Thermal conductivity	35 w/m-k
Coefficient of thermal expansion	8.4×10^{-6}/k
Poisson ratio	0.22

Key properties: High strength and hardness, high temperature stability, high corrosive resistance, high wear resistance.

Applications: Used in electrical industry for insulating parts, in electronics industry as a substrate, protective corrosive coatings and high temperature applications.

3.2 Composite fabrication

For the purpose of fabrication of composite Epoxy resin, used as the matrix material and the hardener (HY951) are mixed in a ratio of 10:1 by weight as recommended. Selection of epoxy is due to its low density (1.1 gm/cc) and low value of thermal conductivity (0.363 W/m-K). Solid alumina particulates are reinforced in the resin to prepare the composites. The dough is then mixed with hardener and is slowly poured into cylindrical glass up to certain depth. Prior to this, glass is first sprayed with thin film of silicone-releasing agent. The purpose of spraying silicon is to safe and easy removing of fabricated composite. The whole thing is then left at room temperature for 25-30 hours for complete polymerization and hardening of polymer composite. Then removing the mold by destroying it to get the sample specimen. The released sample is now available for the testing of various properties.

Fig. 3.5 Images taken during the fabrication of composites

Table3.3: List of composites fabricated for experimental analysis by hand lay-up technique

Sample no.	Composition
1	Epoxy + 3.27 vol %(10.68 wt. %) of filler
2	Epoxy + 7.75 vol %(22.92 wt. %) of filler
3	Epoxy + 13.4 vol% (35.37 wt.%) of filler
4	Epoxy + 26.18 vol %(55.63 wt. %) of filler

3.3 Development of analytical model

A 3-D view of lattice of proposed model showing various spherical particle insides the cube in FCC manner also showing the heat flow direction [58]. Composite material is the combination of all such cubes. Here heat flows from top to bottom.

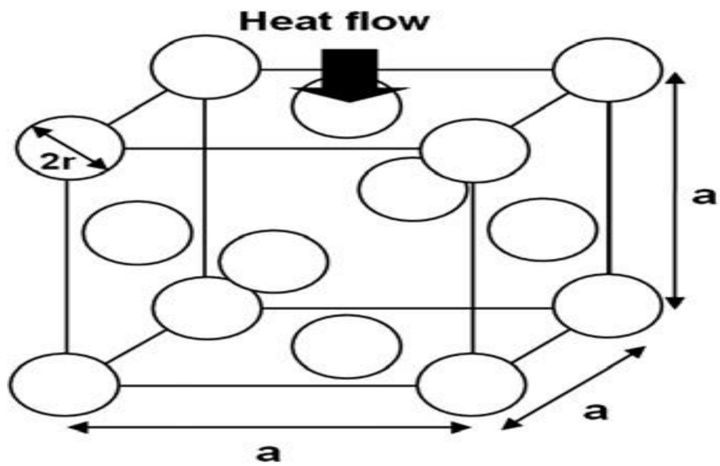

Fig.3.6 3-D view of the analytical model

Symbol meaning:

a = side of cube,

r = radius of particles,

$\emptyset =$ Volume fraction

K_e = epoxy thermal conductivity

K_p = particulates thermal conductivity

Top view of an element shown in figure below which having cube of the side length (a) and spherical particle having radius (r). Top view area is the area of cross-section of the cube for the heat flow as heat is flowing from top to bottom.

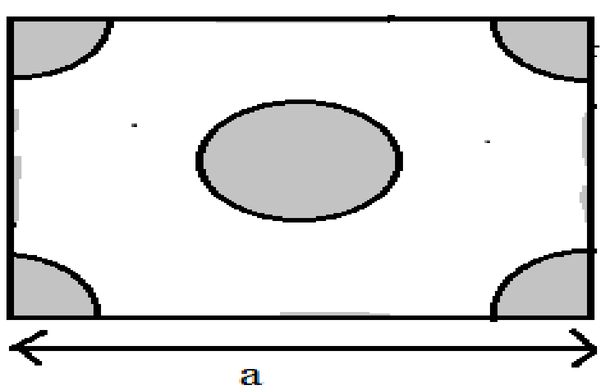

Fig.3.7 Top view of the analytical model

Whereas side view of model looks like, dividing it in the 6 region for ease in calculating total thermal resistance, which is shown in the figure

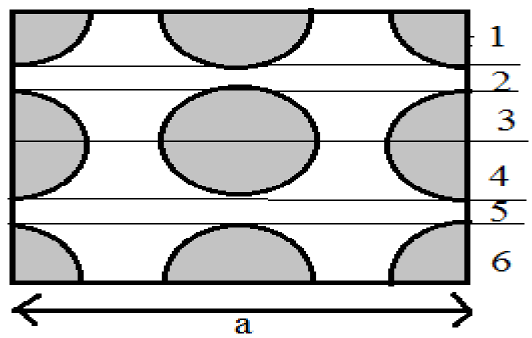

Fig.3.8 2-D view of the analytical model

Now calculating thermal resistance of first region i.e. R_1

Total x/c area of heat flow is a^2.

Particulates area is $2\times \pi r^2$, remaining area = $(a^2 - 2\times \pi r^2)$

Hence considering up to the depth of radius r i.e. region R_1, we can calculate resistance up to that region by integrating thermal resistance formula $\frac{dy}{KA}$.

Assuming Effective thermal conductivity of layer 1 is K^* then from Fourier's law of heat conduction $K^* = \frac{K_e A_e + K_p A_p}{A}$

Now consider a layer of thickness dy at distance y from the top, here equivalent radius is

$x = (r^2 - y^2)$ which is explained in figure

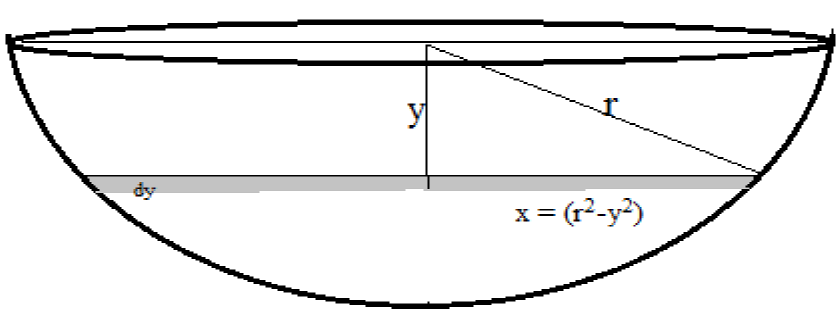

Fig.3.9 cross-sectional view of spherical particulates

$K^{*}\times A = K_e A_e + K_p A_p$

$R_1 = \int_0^r \frac{dy}{K^* A}$

$R_1 = \int_0^r \frac{dy}{K_e A_e + K_p A_p}$

$R_1 = \int_0^r \frac{dy}{K_e(a^2 - 2\pi(r^2 - y^2)) + K_p \times 2\pi(r^2 - y^2)}$

$R_1 = \int_0^r \frac{dy}{K_e a^2 + 2\pi r^2 (K_p - k_e) - 2\pi y^2 (K_p - k_e)}$

$R_1 = \frac{1}{2\pi(K_p - k_e)} \int_0^r \frac{dy}{\frac{K_e a^2}{2\pi(K_p - k_e)} + r^2 - y^2}$

$R_1 = \frac{1}{2\pi(K_p - k_e)} \int_0^r \frac{dy}{b^2 - y^2}$ where, $b = \sqrt{\frac{K_e a^2}{2\pi(K_p - k_e)} + r^2}$

Now from Fourier's law of heat conduction thermal resistance of second layer, $R_2 = \frac{a - 4r}{2K_e a^2}$

Hence total resistance of model,

$R_{total} = R_1 + R_2 + R_3 + R_4 + R_5 + R_6$

Solving equation using the formula $\int \frac{1}{a^2 - x^2} = \frac{1}{2a} \ln \left|\frac{a+x}{a-x}\right|$

$R_{total} = \frac{1}{\pi(K_p - k_e)b} \ln \left|\frac{b+r}{b-r}\right| + \frac{a - 4r}{K_e a^2}$ where $b = \sqrt{\frac{K_e a^2}{2\pi(K_p - k_e)} + r^2}$

3.4 Experimental procedure of thermal conductivity determination

The Unitherm^{TM} model 2022 also known as guarded heat flow meter is used to measure the measure the thermal conductivity of fabricated composite (epoxy filled with alumina particle). For measurement of thermal conductivity, a circular shape of approximately 1 centimeter thick and 3 centimeter diameter sample with various volume fraction of alumina particles are prepared by the hand layup technique. The experimental test is accordance with ASTM E-1530 standard. To get error free and excellent measurement this device has facility to airtight compartment to keep the sample of measurement free from moisture.

Prepared sample are kept in between two polished surface having two different temperature under uniform compressive load. There is a flow of heat from the upper surface whereas lower surface is calibrated to heat flow transducer which will from an axial temperature gradient through the sample under examination of thermal conductivity test. When there is achievement of thermal equilibrium the temperature difference is recorded by temperature sensors, the output heat flow are measured by heat flow transducer. All the data obtained from the experiment is recorded and use it further for the calculation of thermal conductivity.

Fig. 3.10 Experimental setup for measurement of thermal conductivity

To minimize the transfer of heat loss from various edges, sample is surrounded by the guard plte. The complete description of procedure applied during the thermal characteristic measurement of alumina filled with various volume fraction are nicely described below with the help of schematic diagram shown in Fig. 4.3

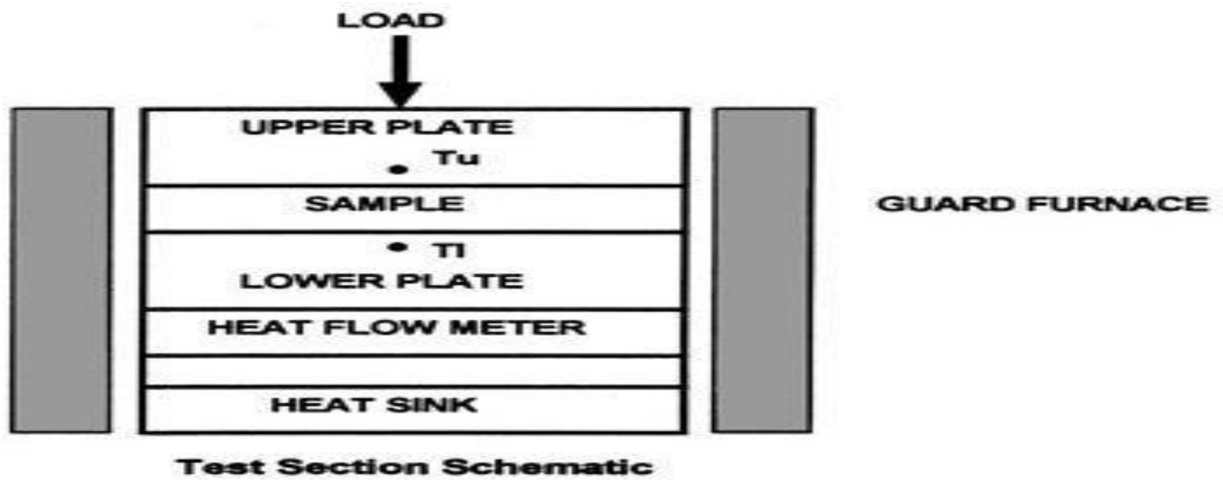

Fig. 3.11 Schematic diagram of experimental setup

Heat flux transducer or heat flow meter is used to measure quantity of heat flow in unitherm™ model 2022. Temperature difference is measured with the help of the temperature sensor used for each plate. By introducing the sample having appropriate and calculated area perpendicular to the heat flow and accurately measured thickness completely end the parameter requirement for calculating the effective thermal conductivity.

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CHAPTER 4

RESULTS AND DISCUSSION

Entire research work is based on the exploration of some novel materials having high thermal conductivity along with low electrical conductivity as it is the basic requirement of our current research for the purpose of use in the field of microelectronics industries for the application in the form of printed circuit board, electronic packaging etc. The possibility of having all these properties in single material is very rare found or even not found. These all properties are possible by fabrication of composite material having ingredients containing some of these properties. In present work as per requirement selected material is epoxy and aluminium oxide as matrix and filler material respectively.

4.1 Results obtained by various method

First we develop a theoretical model for obtain a co-relation or expression for calculating effective thermal conductivity of fabricated composite by hand layup technique having different volume fractions of the filler material. The results obtained by this o-relation is shown in Table

Table 4.1 Results obtained from proposed model

Volume fraction (%)	Effective thermal conductivity(W/m-K)
0(no filler material)	0.363
3.27(a= 8r)	0.61119
7.75(a=6r)	0.8736
13.4(a=5r)	1.3210
26.18(a=4r)	4.84511

From various available co-relations or theoretical model developed by many researcher earlier is used to compare the results obtained from the proposed co-relations. The results obtained by some of these is shown in table along with the co-relations used by them for evaluating the effective thermal conductivity.

Calculation using **rules of mixture [45],**

$$\frac{1}{k_{eff}} = \frac{1-\emptyset}{k_e} - \frac{\emptyset}{k_p}$$

Table 4.2: Results of rules of mixture

Volume fraction (%)	Effective thermal conductivity(W/m-K)
0	0.363
3.27	0.3751
7.75	0.39315
13.4	0.41849
26.18	0.48993

Calculation using **Maxwell model [48]**,

$$K_{eff} = k_e \left[\frac{k_p + 2k_e + 2\emptyset(k_p - k_e)}{k_p + 2k_e - \emptyset(k_p - k_e)}\right]$$

Table 4.3: Results of Maxwell model

Volume fraction	Effective thermal conductivity(W/m-K)
0	0.363w/m-k
3.27	0.3986w/m-k
7.75	0.4514w/m-k
13.4	0.5256w/m-k
26.18	0.7334w/m-k

Calculation using **Lewis and Neilson co-relation [47]**

$$k_{eff} = k_e \left[\frac{1 + AB\emptyset}{1 - BC\emptyset}\right]$$

For spherical particle,

A=1.5, B = $\frac{\frac{k_p}{k_e}-1}{\frac{k_p}{k_e}+1.5}$ and C= 1+$\emptyset\left(\frac{1-\emptyset_m}{{\emptyset_m}^2}\right)$, Where, $\emptyset_m$ = maximum packing fraction of filler

Table 4.4: Results of Lewis and Neilson co-relation

Volume fraction (%)	Effective thermal conductivity(W/m-K)
0	0.363
3.27	0.0.3932
7.75	0.43961
13.4	0.50843
26.18	0.73253

To validate the results obtained from the proposed co-relation developed mathematically under some assumptions in chapter 3 and compare results obtained from various co-existing expression developed by many researchers a laboratory scale experiment is performed by using guarded heat flow meter or Thermal conductivity tester Unitherm™ 2022. The results obtained from the experiment is shown in Table 5.5

Table 4.5 Results obtained from experimental analysis

Volume fraction (%)	Effective thermal conductivity(W/m-K)
0	0.363
3.27	0.534
7.75	0.812
13.4	1.131
26.18	4.237

4.2 Result analysis at constant volume fraction

For analyzing the results obtained from the proposed co-relations, various existing co-relations and experimentally determined values at fixed volume fraction are the following comparison diagrams are drawn separately.

(a) At volume fraction of 3.27% or (10.68 wt. %) of filler

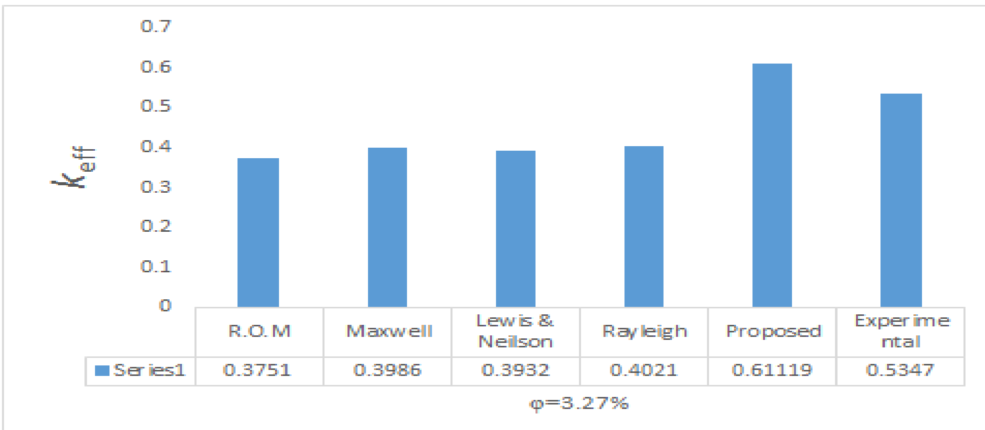

Fig. 4.1 Comparison of results of various co-relation at φ=3.37

(b) At volume fraction of 7.75% or (22.92 wt. %) of filler

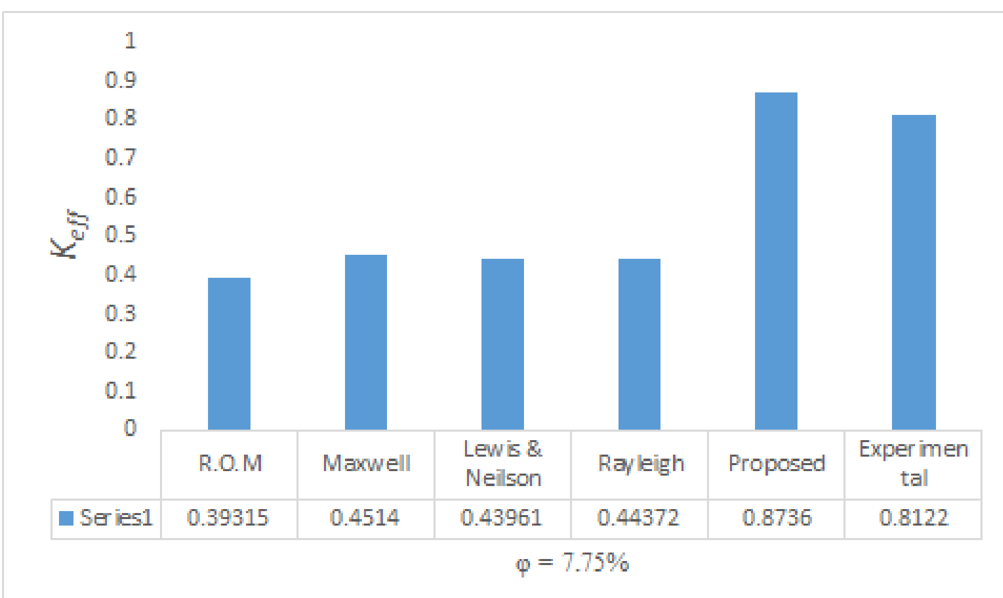

Fig. 4.2 Comparison of results of various co-relation at φ=26.18

(c) At volume fraction of 13.4% or (35.37 wt. %) of filler

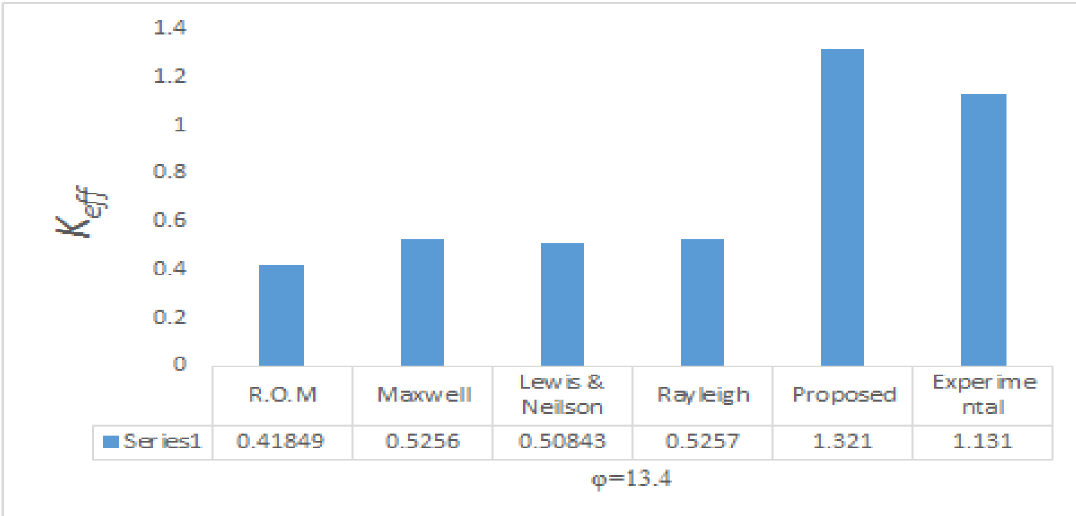

Fig. 4.3 Comparison of results of various co-relation at φ=13.4

(d) At volume fraction of 26.18% or (55.63 wt. %) of filler

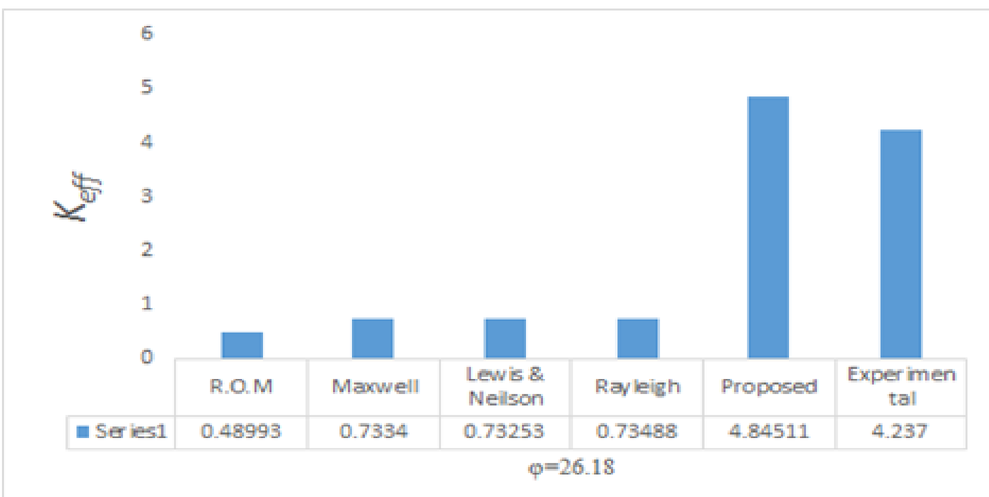

Fig. 4.4 Comparison of results of various co-relation at φ=26.18

Observations obtained from the above all four comparison diagrams concludes that at low volume percentage all conventionally used co-relations gives approximately close results but as there is increase in percentage of filler materials it start under estimating the thermal conductivity values of the fabricated composites.

4.3 Comparison between theoretical and experimental

The variation between experimentally measured values and calculated results from the proposed co-relations shown in Table 4.6

Table 4.6 Comparison between theoretical and experimental value

Volume fraction (%)	Effective thermal conductivity	
	Proposed co-relation	experimental
0	0.363	0.363
3.27	0.61119	0.5347
7.75	0.8736	0.8122
13.4	1.3210	1.131
26.18	4.84511	4.237

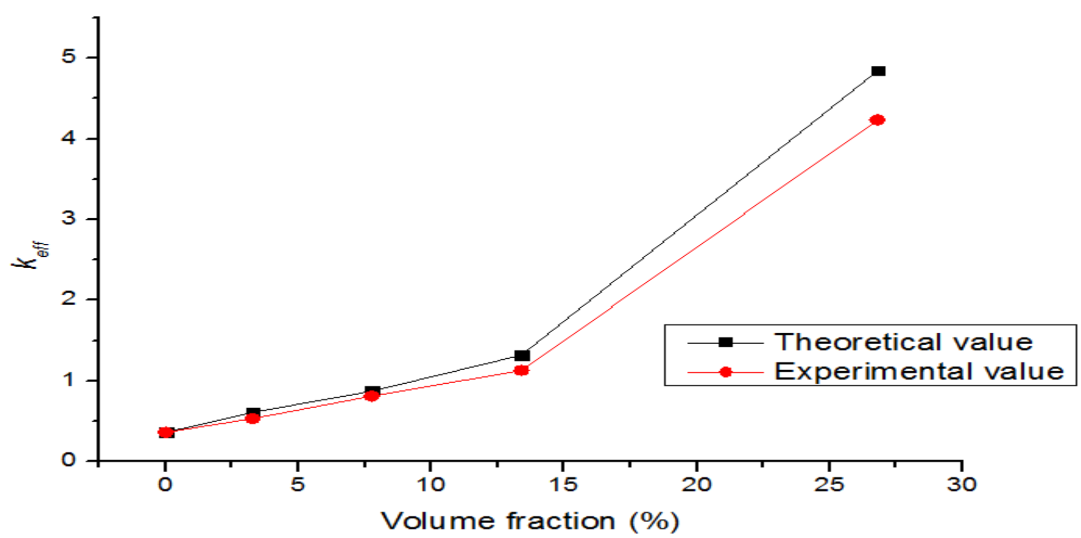

Fig.4.5 Comparison between theoretical and experimental value

From the above graph it can be seen that there is sudden jump in the slope of the thermal conductivity line after some percentage of filler material. These all are happening due to accumulation of filler particles as quantities of these particle are reasonably increased and this led to particulate to particulate heat transfer at the small scale.

4.4 Thermal conductivity increment with volume fraction by proposed co-relation

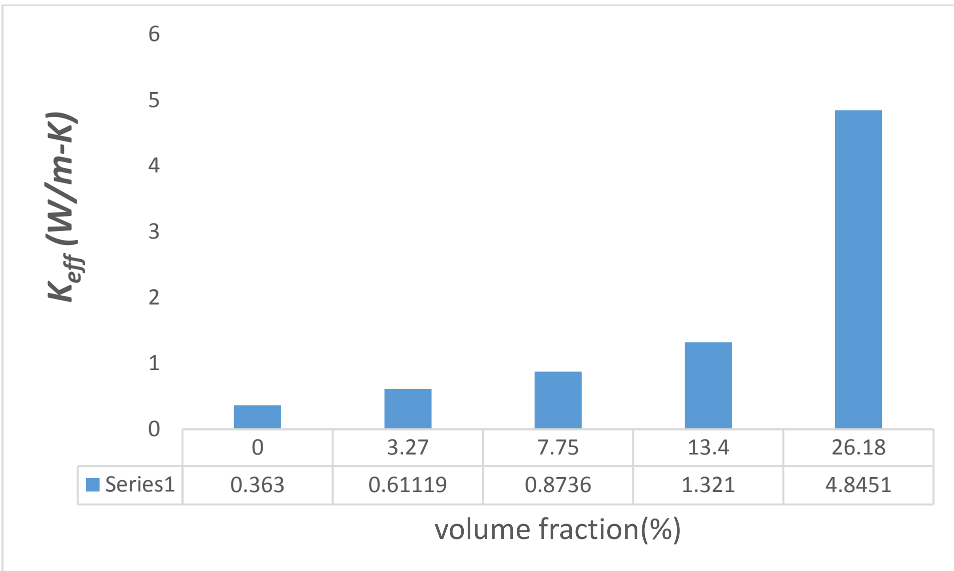

Fig.4.6 Increment in thermal conductivity with filler increment

4.5 Observation of present work

The estimated values of effective thermal conductivity of composites filled with four different (3.27%, 7.75%, 13.4% and 26.8%) filler concentration are shown in figure 4.1, 4.2, 4.3, 4.4 respectively. In each figure the value obtained from different theoretical model and co-relation are shown along with the value obtained from the co-relations proposed in this work. Each of these figure also gives the comparison of theoretical values of effective thermal conductivity against the experimentally measured value.

The following observations are made

1) With increase in the Al_2O_3 content in the composite the effective thermal conductivity improves quite reasonably. This indicates that the incorporation of micro sized Al_2O_3 helps in the enhancing the heat conduction capability of epoxy.

2) The mathematical correlations proposed by previous investigators are found to be underestimating the value of effective thermal conductivity and the deviation from the measured value are quite large.

3) The correlation proposed in this work gives value of effective thermal conductivity for different volume fraction of particulates are found to be in good agreement with the measured value as illustrated in figure 4.5.

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CHAPTER 5

SUMMARY AND CONCLUSIONS

5.1 Brief summary

Research presented on this thesis are summarized as there is a development of mathematical model for very near to exact prediction of thermal conductivity of composites having particulates filled in wide variety of resin commercially available for the need of composite fabrication. The development of this model was based on the assumption that there is only one dimension heat flow, particulates are arranged in disciplined manner as face center cubic arrangement, particles and polymer used in fabrication are homogeneous whereas fabricated composites nature is heterogeneous, there is neglecting of surface contact resistance which is available between particulates and polymeric resin used in the fabrication. This model lead to emerging an expression for calculating thermal conductivity. Detailed explanation about the polymer and particulate used during the research also showing advantages and reason why only this is used in the entire analysis. To validate the results obtained from the proposed model, a very precise experiment was carried using the guarded heat flow meter commonly known as unitherm^{TM} model 2022. Results are also compared by various co-relation derived by researcher for predicting the thermal conductivity.

5.2 Concluded points

These are some noble points concluded from the entire work in search of composites suitable for better heat dissipation.

- ❖ Successful fabrication of epoxy composites reinforced with micro-sized Al_2O_3 particulates is possible by hand lay-up technique.
- ❖ Incorporation of these particles significantly affects the heat conduction capability of neat epoxy.

- ❖ A mathematical correlation has been developed by taking the one-dimension heat conduction across the sphere-in-cube three-dimensional physical model to estimate the effective thermal conductivity.
- ❖ It is seen that the effective thermal conductivity of such particulates filled composites is a function of filler content and of the intrinsic properties of filler and matrix materials.
- ❖ The correlation is validated by conducting laboratory scale measurement of effective thermal conductivity of these composites and then by comparing the test result.
- ❖ It is found that the results obtained with the addition of 3.27%, 7.75%, 13.4% and 26.8% the thermal conductivity increases by 47.3%, 123%, 211% and 1067% respectively.
- ❖ It is also found that the results obtained from proposed co-relation are in very good agreement with the experimentally measured value.
- ❖ Composites fabricated with alumina particles having reasonably enhanced thermal conductivity are suitable for the use as printed circuit board, electronic packaging and encapsulation purposes.

5.3 Scope for the future work

There is lots of work remaining for future researchers for getting the best results in the practical application of these alumina filled composites. There is requirement of explore the other aspects of thermal behavior. These are some recommendation from my side --

- ➢ Try to investigate the results for the variation in shape and size of the particulates.
- ➢ Try to find out the effect of thermal contact resistance between particulates and polymer used for fabrication of composites.
- ➢ Exploration of new polymers along with particulate having better thermal conductivity along with low electrical conductivity.

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